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(54) Title: **SLURRY OF AGRICULTURAL FIBRES AND ITS PRODUCTS**

(57) Abstract: Environmental friendliness and biodegradability are important considerations of disposable materials, be it packaging or paper, in order to minimize environmental damage. The present invention relates to a method of producing environmental friendly and biodegradable products using mechanically processed agricultural fibres and rightly formulated adhesive. In particular, mechanical treatment takes place in a pressure vessel under hydrothermal conditions using superheated steam. Treated agricultural fibres are mixed with stoichiometric amount of adhesive, through vigorous agitation, a well furnished agricultural fibre slurry is obtained and ready to be fed into manufacturing process to produce, in this invention, moulded shape bodies and paper liners. Unlike synthetic resin, the said adhesive formulation is biodegradable and made of natural renewable resources. The said adhesive has self-retention property. The agricultural fibre slurry can be easily made into moulded products and paper liners using commonly available processes.

SLURRY OF AGRICULTURAL FIBRES AND ITS PRODUCTS

The present invention relates to a method of producing environmental friendly products using hydrothermally treated agricultural fibres and a natural adhesive formulation. In particular, the hydrothermally processed agricultural fibres and the natural adhesive are thoroughly mixed to produce required aqueous slurry that is ready to produce moulded shape bodies and paper liners using relevant manufacturing processes.

BACKGROUND

Recent effort in developing environmental friendly and biodegradable packaging products has lead to some interesting results. Among a number of environmental friendly packaging alternatives, moulded pulp packaging, starch-based packaging and photodegradable plastic packaging materials are emerging to be commercially viable alternatives to conventional plastic packaging products. However, if we were to look at the entire cycle of manufacturing process from the production of raw materials to the manufacturing of packaging products, each of these materials has its own unique environmental weaknesses and much improvement is desired to enhance its environmental competitiveness.

Taking moulded pulp packaging for example, paper is more biodegradable than plastics, however paper is far more polluting to the environment to manufacture, not to mention chlorinated pulp bleaching process. Looking at business economy, moulded pulp packaging is an expensive alternative to plastic packaging, especially when virgin pulp or thoroughly processed recycled pulp is used as raw materials.

Starch-based packaging is a rapid developing alternative due to the highly machineable characteristic of starch-based materials, however, its edible nature has limited its applications. Photodegradable plastic is the closest to ordinary plastic material in terms of manufacturability. However, market acceptance and large-scale market penetration would not take place until the photodegradability of this kind of plastic materials proves to be as claimed, and its source of plastic resin does not depend on petroleum only.

Recent efforts in developing biodegradable packaging materials have resulted in published formulations and pilot scale manufacturing of agro-based packaging products. This agro-based packaging is made of agricultural fibre residues which is otherwise incinerated or landfilled, starch and thermoplastic resin as binding agent and inorganic components such as calcium carbonate as filler. Of these agro-based materials, agricultural fibres such as rice husk and rice straw are fillers and their presence enhances the binding power of starch, hence improves mechanical properties of final packaging products. It

is not incorrect to comment that such an agro-composite formulation is of agro-plastic by nature, and usage of agricultural fibres is minimum. The invention of agro-composite provides a value-add solution to discarded massive fibrous residue, and serves to reduce consumption of wood or non-wood pulp. However, as an alternative to environment-damaging packaging material, it has only addressed the issue in partial. Besides, this agro-composite formulation dictates an expensive alternative due to heavy usage of thermoplastic and starch in the formulation. Typically, such an agro-composite comprises of 20-30% of agricultural fibre, 30% to 70% of binding agent and the rest is made up of functional additives such as sizing, grease barrier and wax, etc (weight ratio).

What remains challenging and yet to be addressed is how to formulate an agro-based composite containing agricultural fibres as the major component, while the binding agent and auxiliary chemicals as minor. Such an agro-based composite would be an ideal environmental friendly packaging solution, and it would also guarantee the cost competitiveness of such an agro-based composite formulation.

Solutions to the above question will emerge from well understood key issues of (i) agricultural fibre treatment; (ii) adhesive formulation; (iii) coordinated role of treated agricultural fibre and adhesive, and existing manufacturing technology to work with such a formulation. Treatment applied to agricultural fibre refers to chemical-free treatment serving to activate fibres surface in order to work with the formulated adhesive. If appropriate existing manufacturing technology for moulded shape bodies and paper liners is used, such invented agro-composite material would provide a significant environmental friendly solution with guaranteed cost competitiveness.

What then should be the appropriate manufacturing technology to tap on? Moulded pulp manufacturing is a rather matured manufacturing process. It is designed to work with low consistency pulp slurry, and through mechanical dewater and thermal moulding to give moulded shape bodies. Now then, situation seems to be clear as to how to take advantage of moulded pulp manufacturing technology and through modification to produce moulded agricultural fibre shape bodies such as packaging. Similar analogy applies to the making of paper liners by adopting existing paper manufacturing process to produce agricultural fibre based paper liners from well balance application of treated agricultural fibre and adhesive formulation. Case studies of moulded pulp and paper manufacturing processes revealed that they are made from similar low consistency pulp slurry. In order to make use of the existing manufacturing technology, low consistency agricultural fibre slurry furnished with adhesive should be used as the feedstock.

Up to this point, the intention of the present invention is evident that is to provide a generic recipe for making of moulded shape bodies and paper liner from low consistency slurry containing treated agricultural fibres, hereinafter known as AgroFibre, as the major component.

German patent, DE19949975, discloses a fabrication method for insulation and construction panels and molded parts uses dry lignocellulose fibres moistened in several stages and powderized binding agent added during moistening process. The panels/parts are molded from biodegradable materials, e.g. lignocellulose fibres and natural starch-containing binding agents. The dry fibres are moistened in one or more stages by the addition of water, to 15% to 50%, with a moisture content of 13% to 33%. The powderized binding agent is added to the fibres after at least one moistening stage. The surfaces of the formed panels or parts are finished by spraying with water or water-binding agent suspension, before hardening. The panels/parts are finished in a hot press or a drier outside the press. The binding agent may be a starch-containing potato material, and the fibre material may be wood fibres, or fibres of wheat or barley straw, oil seed rape straw, hemp or flex, or a mixture of these.

Chinese patent, CN1312158 by GUO KANGQUAN, YANG ZHONGPING and ZHU XINHUA describes a production method of fibre composite moulded product. The invention uses soft thin-layer product as facing material and uses glued fibre as core layer material, after both are compounded, they are hot-pressed and formed in the die equipment, then undergone the processes of die-cutting and surface treatment to obtain the invented fibre composite moulded product. It can produce thin-wall shaped product with large depth and large obliqueness.

US patent, US5679145, of Khashoggi E Ind (US) discloses compositions, methods, and systems for manufacturing articles, particularly containers and packaging materials, having a starch-bound cellular matrix reinforced with substantially uniformly dispersed fibres.

Chinese patent CN 1230386 describes a method of producing dinnerware made from plant fibre. This patent uses plant fibres and adhesive with almost no water content. The mixture is of high viscosity and presents as a lump to be poured into a mould or die to be heated under significantly high temperature and pressure.

Chinese patent CN1257089 discloses a production method of plant fibre foamed packaging material. The composition includes plant fibres, foaming agent and additives of starch to be heated in the mould of the desired shape.

US patent US5849152 (PCT/EP95/00285) describes a process for the production of shaped bodies from biologically decomposable material using a viscous mass containing fibre, water and starch, which is baked in a baking mould to produce a shaped body. The amount of starch is 15 wt % to 200 wt % the dry weight of the fibre material.

SUMMARY OF THE INVENTION

The present invention discloses a formulation of using low consistency AgroFibre slurry for manufacturing environmental friendly and biodegradable moulded products and paper liners.

The AgroFibre slurry consists of adhesive hereinafter known as AgroBinder, and hydrothermally processed agricultural fibres, AgroFibre.

An object of the present invention is that the adhesive, AgroBinder, is a starch-based, alkaline and viscosity stable adhesive that is a natural polymer and is biodegradable on its own.

Another object of the present invention is to eliminate the use of chemical treatment of agricultural plant fibres so that polluting effluent can be totally avoided while increased affiliation with adhesive, AgroBinder, is achieved. In the present invention, agricultural fibres including empty fruit bunches (EFB) of oil palm, coconut coir, bagasse and rice husk are processed through mechanical means under hydrothermal conditions using live steam to produce AgroFibre. Technical specification of AgroFibre affects the performance of AgroBinder and functional chemicals such as sizing, wet strength and grease barrier..

According to another object of the present invention, AgroBinder is self-retaining to AgroFibre and its formulation allows functional chemicals to impart sizing, grease barrier and wet strengthening properties to the finished AgroFibre products, be it moulded packaging or paper liner.

According to another object of the present invention, AgroFibre is obtained by a chemical-free treatment in which agricultural fibres are processed in a hydrothermal digestion tank using superheated steam at an operating temperature between 150 - 250 deg C and pressure between 0.3 MPa to 3 MPa for a residence time of 10 to 120 min.

According to yet another object of the present invention, the moulded shape bodies and paper liners are manufactured using AgroFibre slurry as the moulding material. The AgroFibre slurry consists of thoroughly reacted AgroFibre, water, AgroBinder and optional functional additives. During the moulding process, the AgroFibre slurry is mechanically dewatered and thermally cured to give the required shape and mechanical properties. The weight percentage of AgroFibre in the final product is in the range of 90 to 97% with AgroBinder and functional additives taking up the rest of the composition. Another aspect of the formulation of AgroBinder allows the manufacturing of moulded AgroFibre shape bodies from low consistency AgroFibre slurry using the modified moulded pulp manufacturing process.

AgroBinder is made of the following compositions: water, starch carrier, ungelatinized starch, modified starch, green-bond developing agent and caustic. AgroBinder is manufactured based on the following process: starch

carrier phase at pH between 12-14 is cooked to give gelatinized starch, to which ungelatinized starch is added under continuous heating and stirring conditions. Next, green-bond developing agents are added to the above mixture. Mixing is continued for at least 30 minutes until a homogenous gelatinized mixture is obtained.

According to yet another aspect of the invention, the formulation of AgroBinder also makes paper-making possible from AgroFibre without going through intensive chemical pulping process, hence establishing a pollution-free paper-making approach.

DESCRIPTION OF DRAWINGS

Figure 1 illustrates the chemical-free, multi-stage treatment process of agricultural fibre under hydrothermal condition via mechanical means to produce required quality of AgroFibre.

Figure 2 shows the preparation of low consistency AgroFibre slurry ready for the manufacturing of moulded shaped bodies and paper liners.

PREFERRED EMBODIMENT OF THE INVENTION

A preferred embodiment of the present invention uses empty fruit bunches (EFB) of oil palm that are residues of the crude palm oil extraction process. EFB of oil palm contains huge amount of fibres that are long, strong and easy to extract. EFB fibres, one of the most abundant types of agricultural fibrous residues, are produced as byproducts and discarded in palm oil mills. Taking Malaysia for example, some 40 million tons of EFB is burnt and landfilled at cost every year.

EFB fibres [20] are fed into a screw press [21] for further removal of its oil content. The screw press [21] applies pressure to the EFB fibres [20] to squeeze the residual oil [25] and water from the EFB fibres [20]. The residual oil [25] is an additional income for the palm oil mill. The de-oiled and dewatered EFB fibres are then fed into a hydrothermal digestion tank [22]. The hydrothermal digestion tank [22] is supplied with superheated live steam. The operation condition of the digestion tank [22] is kept at a temperature between 150 deg C to 250 deg C, with a pressure of 0.3MPa to 3 MPa. The EFB fibres in the hydrothermal digestion tank [22] are continuously agitated for 10 to 120 mins and preferably 30 to 60 mins under the set pressure and temperature. The agitation action increases the solvation effect of the superheated steam and facilitates delignification of EFB fibres. The hydrothermally treated EFB fibres are discharged from the digestion tank [22] and then cut by a cutter [23] to the desired length between 1 to 50 mm and preferably 10 to 30 mm. Next, the cut EFB fibres are fed into a disc refiner [24] to produce the AgroFibre [30]. The refiner [24] is similar to those found in the pulp industry such as a disc refiner. The AgroFibre [30] at the output of the refiner [24] has high moisture content. It can be used immediately in an in-line

process or if storage of AgroFibre is required, then drying will be required to prolong its shelf-life. No chemical is used during the hydrothermal processing of the EFB fibres and hence no polluting discharge is generated. The exhausted steam can be condensed into water and recycled accordingly.

The AgroBinder disclosed in the present invention is a starch-based adhesive formulation. AgroBinder can be carrier type, no-carrier type and carrier-no-carrier type depending on the type of starting materials and process applied to the manufacturing of AgroBinder.

The composition of AgroBinder comprises of starches as binding agent and retention aid, green-bond developing agents, waterproofing resin and caustic. The binding agent of AgroBinder is starch that is gelatinized as heated. Other components including caustic, green-bond developing agents and waterproofing resin are auxiliary agents that enhance the properties of starch.

Native and/or modified starch is employed to give the binding power of AgroBinder. Native starch is an economical source of starch, however, preparation can be lengthy and equipment-intensive. Modified starch can be mechanically and chemically modified. Sensitivity to the source of starch including tapioca starch, corn starch, sorghum starch and potato starch is not observed. The advantage of using modified starch comes from its enhanced properties such as improved solubility, paste stability, viscosity stability, enhanced adhesivity and resistance to freeze-thaw degradation and microbial activity, higher solid content and controlled electron charge density, etc. It eases the manufacturing process and reduces one-time manufacturing investment. A variety of chemically modified starches are suitable for the preparation of AgroBinder. This includes typically cross-bonded starch, oxidized starch, cationic starch modified with tertiary amino or quaternary ammonium compounds and starch-derivatives which is the choice of required finishing properties of the starch adhesive.

Modified starch used in the preparation of AgroBinder are typically those of Rouquette, Cerestar, Kalamazoo, etc. Unmodified starches that are used in the non-carrier phase include potato starch, corn starch and tapioca starch. Dextrins have been proved fit as carrier and it can be prepared or purchased.

Retention aid is a crucial component of AgroBinder. Many types of retention aids are available. The preferred choice of the present invention is modified starch, typically as oxidized starch. It also forms part of carrier phase of the adhesive formulation. Its presence enables the starch adhesion, AgroBinder, to retain to AgroFibre during the preparation of AgroFibre slurry.

Caustic is introduced in the form of aqueous solution of sodium hydroxide or potassium hydroxide. The dosage and point of application affect the gelatinization temperature of the starch. Besides, it allows a better and faster bonding development during manufacturing process.

Green-bond developing agent usually consists of a group of compounds, typically including polyhydroxy compounds and boron-containing compounds.

It is to increase adhesive viscosity and viscosity stability via the active hydroxyl group of polyhydroxy compounds and oxygen-boron bonds of boron-containing compound. This provides the required viscosity and viscosity stability of starch-based adhesive formulation. The presence of boron-containing compound also provides buffering effect to the viscosity and alkalinity stability of adhesive. Coexistence of polyhydroxy compounds and boron-containing compounds provides a fast-developed tackiness which allows paper liner and moulded shape bodies to form at acceptable speed in the production process.

If additional water repellency is required for AgroBinder, water resistant resins can be added to the starch adhesive formulation. Typically, they are cross-linking agents that react with available hydroxyl groups of starch. A number of compounds including polyvinyl alcohol and polyvinyl acetate, methylcarboxylate cellulose and glyoxal meet the required performance. These cross-linking agents reduce the hydrophilic nature of starch-based adhesive by screening hydrophilic sites through developed hydrophobic moieties.

EXAMPLES

Weigh 65 gram of water, to which 9 gram of a mixture of native (cold water soluble) and cationic starch are slowly added. The mixture is stirred in the primary mixer for more than 10 minutes and followed by dropwise addition of aqueous solution of caustic soda to attain a pH of 12-14. The resultant mixture is stirred at 65-75 deg C until a yellowish homogenous gel is obtained. Next, 155 ml of water and 1.6 gram of $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ are introduced to the above homogenous gel, and followed by the addition of 40 gram of native starch. The resulting mixture is stirred at 35-45 deg C for 30 minutes. When water repellency is required, water-resistant resins and other functional agents can be introduced to the above homogeneous mixture for a furnished formulation catered for specific application.

If ordinary native starches are used, jet cooker and live steam are recommended for the preparation of AgroBinder to facilitate starch dissolution. Potassium hydroxide can be used instead of sodium hydroxide. Boric acid and other boron-containing compounds are alternatives to borax used in the preparation of AgroBinder.

Potato starch, tapioca starch and cornstarch are suitable for the above preparation and the resulted AgroBinder shows similar performance in slurry application for making of moulded shape bodies and paper liner.

The preparation of AgroFibre slurry is described next. AgroFibre [30] is added to water [50] in a mixing tank [65] and dispersion of AgroFibre is facilitated via vigorous agitation. Preferred consistency of AgroFibre slurry is 2-6 % of (weight percentage) AgroFibre to water ratio. AgroFibre swells under solvation effect of water and a higher consistency leads to poor solvated AgroFibre. AgroBinder [10] is then added to the mixture in the mixing tank [65] and continuous agitation is carried out. Due to the self-retention property,

AgroBinder is attached to the AgroFibre. Mixing is carried out for 10 to 60 min to give a well furnished AgroFibre slurry [80]. Functional additives [51] such as sizing and wet strength, if required, are then added to the mixture. Vigorous agitation should be avoided to ensure better retention of functional chemicals to AgroFibre. The solid content of the AgroFibre slurry preferably comprises 80 to 95 wt% of AgroFibre, 5 to 15 wt% of AgroBinder, and less than 5 w% of other additives. It is evident that the principal component of the AgroFibre slurry is AgroFibre - the mechanical hydrothermally processed agricultural fibres.

The said AgroFibre slurry [80] can be diluted by adding even more water to produce low consistency slurry (0.1% to 3%). Slurry consistency of 0.3 to 1.5% is preferred if moulded shaped bodies are to be produced using process similar to moulded pulp manufacturing. Moulded AgroFibre shape bodies are formed through vacuum dewater, followed by mechanical dewater and thermal curing at pressure of 0.2 to 2 MPa and temperature between 120 to 200 deg C. Heating under applied pressure facilitates the curing process and strengthens the mechanical properties of the moulded shaped bodies. The cured adhesive binds all the plant fibres together forming a strong moulded shape body.

In another preferred application of the said AgroFibre slurry is in making of paper liners. Low consistency AgroFibre slurry is fed into a paper making machine the way low consistency pulp is used. Low consistency slurry ensures good formation and uniformity in the produced paper liners.

CLAIMS

1. AgroFibre slurry comprises mechanically processed agricultural fibres, AgroFibre, and a natural adhesive formulation, AgroBinder; the said mechanical processing of agricultural fibres is chemical-free in which the agricultural fibres are mechanically pressed, digested under hydrothermal conditions, cut and refined; the said natural adhesive formulation is carrier, no-carrier and carrier-no-carrier type which is starch-based, alkaline and viscosity stable that is biodegradable, self-retaining and water resistant.
2. The AgroFibre slurry as claimed in claim 1 wherein the agricultural fibres are digested at hydrothermal conditions using live superheated steam and at temperature between 150 to 250 deg C, and pressure between 0.3 to 3 MPa for a residence time of 10 to 120 min;
3. The AgroFibre slurry as claimed in claim 1 wherein the natural adhesive, AgroBinder, comprises water, starch carrier, ungelatinized starch, modified starch, green-bond developing agent and caustic.
4. The AgroFibre slurry as claimed in claim 3 wherein AgroBinder is manufactured by cooking starch carrier phase at pH between 12–14 to give gelatinized starch, and to which ungelatinized starch is added under continuous heating and stirring; green-bond developing agents are subsequently added and mixed for at least 30 min to obtain homogenous gelatinized mixture.
5. The AgroFibre slurry as claimed in claim 4 wherein AgroBinder is self-retaining to AgroFibre due to the addition of retaining aid, preferably oxidized starch, to the AgroBinder formulation.
6. The AgroFibre slurry as claimed in claim 5 wherein caustic, preferably in the form of aqueous solution, is added to the AgroBinder formulation.
7. The AgroFibre slurry as claimed in claim 6 wherein green-bond developing agent, preferably boron-containing compound, is added to the AgroBinder formulation.
8. The AgroFibre slurry as claimed in claim 7 wherein functional additives such as sizing, wet strength and grease barrier agents are added to the slurry to enhance functional performance as options for specific applications.
9. The AgroFibre slurry as claimed in claim 1 is obtained by thoroughly mixing AgroFibre with AgroBinder and water to form aqueous slurry.

10. The AgroFibre slurry as claimed in claim 9 is diluted with water to low consistency slurry between 0.1 to 3% as the feedstock for the manufacturing of moulded shape bodies and paper liners.
11. The AgroFibre slurry as claimed in claim 10 wherein vacuum forming and thermal curing process is used to manufacture moulded shape bodies.
12. The AgroFibre slurry as claimed in claim 11 wherein low consistency pulp is substituted with low consistency AgroFibre slurry for the manufacturing of paper liners.

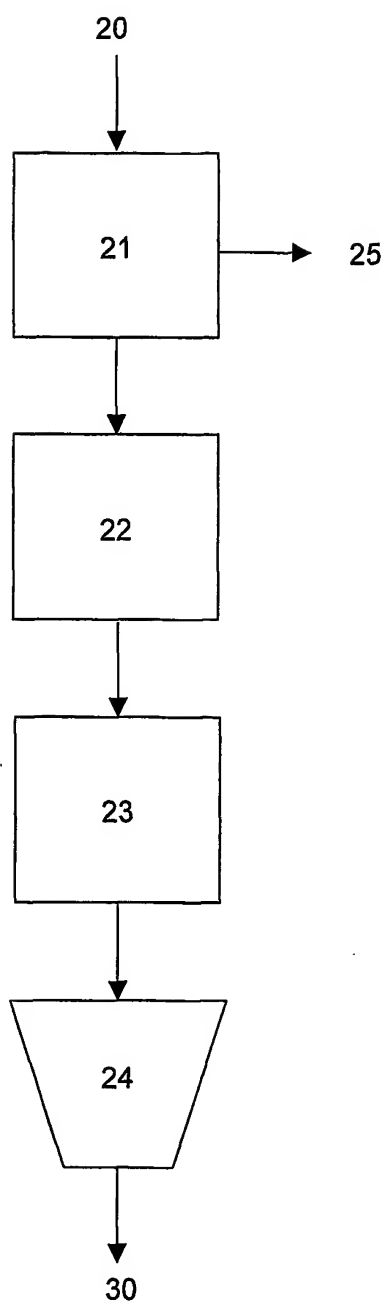


Figure 1/2

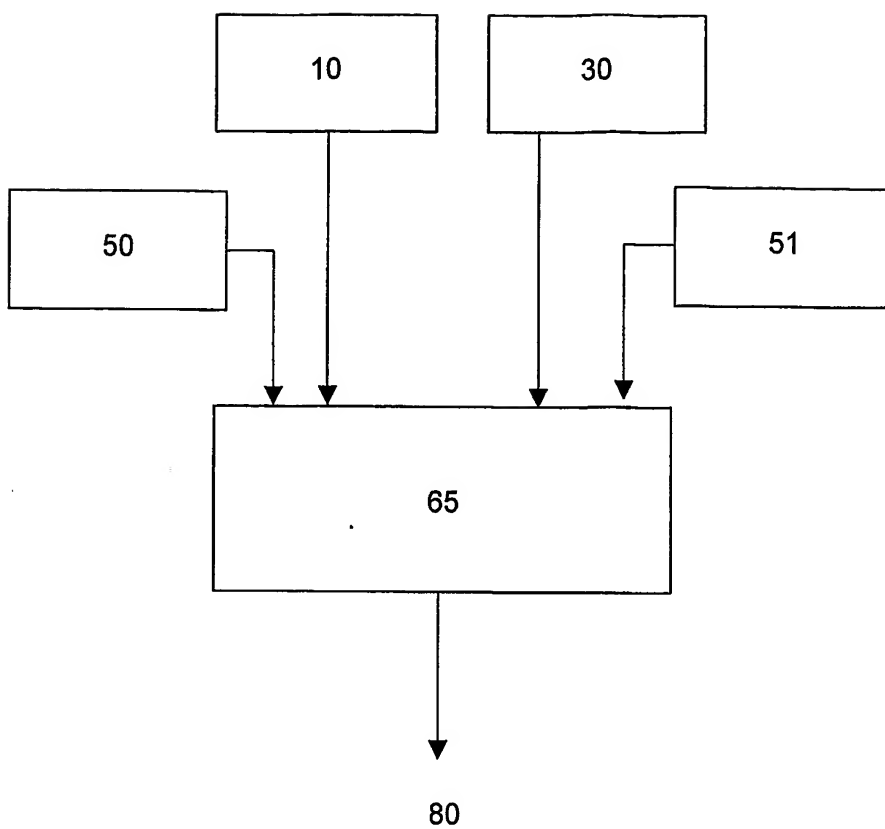


Figure 2/2

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 D21H11/12

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 D21H

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the International search (name of data base and, where practical, search terms used)

EPO-Internal, PAJ, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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X	US 5 656 129 A (GOOD DAVID BENARD ET AL) 12 August 1997 (1997-08-12) claims 1-38	1
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A	DE 199 49 975 A (UNIV DRESDEN TECH) 31 May 2001 (2001-05-31) the whole document	1-12
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☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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"&" document member of the same patent family

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Name and mailing address of the ISA

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INTERNATIONAL SEARCH REPORT

Internatl .pplication No
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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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